DOI 10.1007/s10717-014-9645-7
Glass and Ceramics, Vol. 71, Nos. 5 – 6, September, 2014 (Russian Original, Nos. 5 – 6, May – June, 2014)

UDC 666.1

CALIBRATION OF QUARTZ GLASS TUBES BY VAPOR PHASE DEPOSITION

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Translated from *Steklo i Keramika*, No. 5, pp. 33 – 35, May, 2014.

The results of work on the stabilization of the wall thickness of quartz tubes by means of external vapor-phase deposition during high-temperature hydrolysis of silicon tetrachloride are presented. The technological parameters of deposition and sintering of a porous layer are determined. Defect-free layers of glass of thickness greater than 0.2 mm with the possibility of lowering the circumferential thickness variation of the tubes from 0.2 to 0.05 mm are obtained.

Key words: quartz tubes, circumferential thickness variation, wall thickness calibration, vapor-phase deposition, sintering.

A modified method of chemical vapor-phase deposition MCVD is the most versatile of all known methods of fabricating light guides from quartz glass. Only its unique technological possibilities make it possible to fabricate optical fibers for solving the most diverse technical problems.

The main initial components, whose quality determines the optical and mechanical properties of light guides, in MCVD technology are tubes made from quartz glass. One of the most important technical specifications for these tubes is that their geometrical parameters, especially the wall thickness, must be stable.

For MCVD technology the most cost-acceptable quartz-glass tubes manufactured by the continuous formation from melt of the mineral raw materials possess two main drawbacks:

– presence of micro-nonuniformities of the chemical composition, which results in light guides with low strength;

– large variation of the wall thickness over the circumference, which disrupts the concentricity of the core relative to the outer diameter of the fiber and increases the intermode dispersion of multimode light guides.

The first drawback can be eliminated by depositing on the outer surface of a tube a thin layer of ultrapure quartz glass by means of outer vapor-phase deposition (OVD) [1]. If the tube is not turned during the deposition process, then the wall thickness variation along the circumference of the tube can be decreased [2].

The patents cited in the references contain only extremely limited information about these processes. For this reason the aim of the present work was to pick process parameters (preliminary heating temperatures of the tube, deposition and sintering regimes for the layer) ensure that the glass layer deposited on the outer surface of a tube is defect-free and the variation of the wall thickness is reduced.

EQUIPMENT, INITIAL MATERIALS AND METHODS OF INVESTIGATION

The experiments were performed on the thermo-mechanical stand (Heat Way Company) intended for the MCVD process. Upgrading adapted the stand to the OVD process (Fig. 1).

The OVD burner formed three concentric streams: a mixture of $SiCl_4$ vapor with oxygen at the center followed by a circular stream of screening dry oxygen and a hydrogen flow on the outside. The vapor-gas mixture ($SiCl_4 + O_2$), oxygen and hydrogen are passed through filters in order to remove particles larger than 1 μm. The oxygen contained no more than 10^{-4} vol.% moisture, and the total mass content of impurity metals in the silicon tetrachloride did not exceed 0.02%. The vapor-gas mixture was formed by bubbling dry oxygen at flow rate 2 or 3.5 liters/min through liquid silicon tetrachloride at temperatures 20 – 21°C. The flow rate of the

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TABLE 1. Geometric Parameters of Tubes

Tube type	Outer diameter, mm	Wall thickness, mm	Thickness variation, mm
A	20 ± 0.05	2 ± 0.02	0.04
B	20 ± 0.05	2 ± 0.10	0.20

screening oxygen was 1 liters/min and the flow rate of hydrogen about 5 liters/min.

An oxygen-hydrogen burner was used to heat the tubes and sinter the porous layers. A Promin' optical pyrometer was used to determine the temperature of the tube, taking account of the corrections for the emissivity of quartz glass.

In the process of heating and deposition of porous layers the tube as turned at the rate 50 rpm. To eliminate the circumferential variation of the wall thickness glass was deposited on one side without turning the tube. During sintering of the porous layer the rotation rate of the tube was 25 rpm.

The outer diameter of the tube was monitored with a slide caliper and the thickness of the wall at two ends was measured with dial gauge with 2 μm scale division. The circumferential thickness variation was determined by performing ten measurements of the wall thickness at points uniformly distributed around the circumference.

Quartz tubes fabricated by means of continuous formation during electrosurfacing with grit in a hydrogen medium were used in this work. The geometric dimensions of meter-long tubes are presented in Table 1.

The main difference between the tubes was the circumferential thickness variation, equal to 0.04 and 0.2 for type A and B tubes, respectively. The orientation of the azimuthal distribution of the wall thickness remained over the entire length of the type-B tube, which made it possible to stabilize the thickness of such tubes by depositing quartz glass on one side. The tube is not rotated, and it lies with the thin wall facing the flame of the burner. After the deposited layer is sintered an abrasive wheel is used to cut off process tubes to which the calibrated tube is welded.

The thickness of the wall with the glass layer was measured just as for the initial tubes at 10 points uniformly distributed over the circumference at distances 25 – 30 mm from the ends of the tube.

RESULTS

The number of silicon dioxide particles deposited on the surface of a tube by means of thermophoresis is proportional to the deposition time or inversely proportional to the burner velocity *v*. Regimes for obtaining a prescribed thickness of the tube can be picked on the basis of experimental data on the linear dependence of the thickness of the glass layer in one pass of the burner on $1/v$ (Fig. 2).

Before depositing the porous layer the tube was heated by a burner moving at 2.5 mm/sec in order to burn out or-

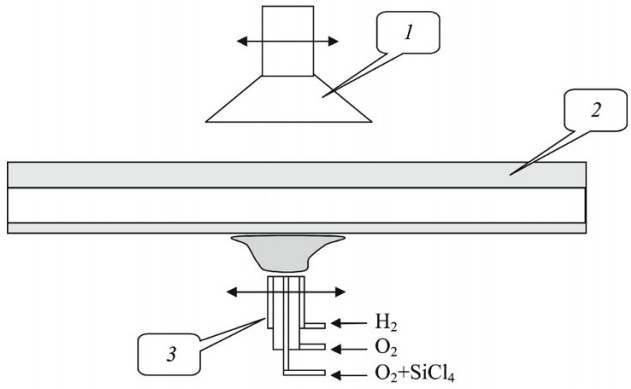

Fig. 1. Schematic diagram of the experimental OVD setup: *1*) exhaust ventilation; *2*) quartz tube; *3*) three-zone oxygen-hydrogen burner.

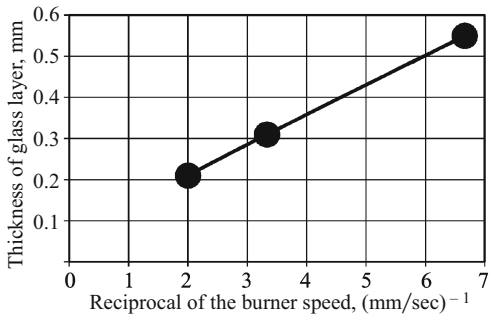

Fig. 2. Thickness of the glass layer deposited on a rotating type-A tube versus the reciprocal of the burner velocity with oxygen flow rate through the evaporator with $SiCl_4 \approx 3.5$ liters/min.

ganic impurities and dissociate inorganic dust particles. The presence of such nonuniformities leads to the formation of a mass of bubbles on the tube surface when the porous layers are sintered. The temperature 1350°C was found to be optimal for this process. More intense heating shrinks the tube, and if the temperature is reduced, the inorganic impurities do not dissociate completely and individual bubbles form in the boundary layer between the deposited glass and the base.

The scan speed of the burner during the sintering of the porous layers, whose thickness decreases by a factor of 2.5 – 3, also affects the quality of the boundary layer. The optimal conditions for their transformation into a glassy state (burner scan speed and the heating temperature) are determined by the thickness of the deposited porous layer. For example, for thickness no greater than 3 mm good quality of the glass and boundary layer is achieved at temperature 1350°C and burner scan speed 0.2 mm/sec. At scan speeds above 3 mm/sec gases are blocked in the porous layer, which destroys the transparency of the boundary layer.

The experimental results on the $1/v$-dependence of the maximum thickness of the quartz glass layer with uniform deposition on a type-A tube (Fig. 3) in one scan of the burner makes it possible to choose the deposition rate so as to de-

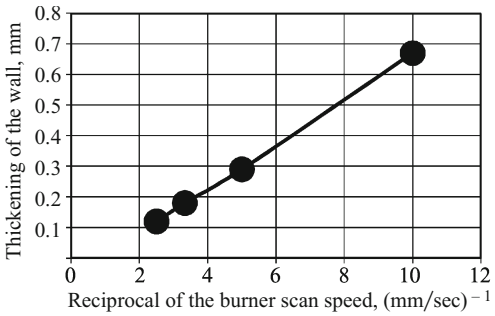

Fig. 3. Maximum thickness of the glass deposited on a non-rotating type-A tube versus the reciprocal of the burner scan speed with oxygen flow rate through the evaporator with $SiCl_4 \approx 2$ liters/min.

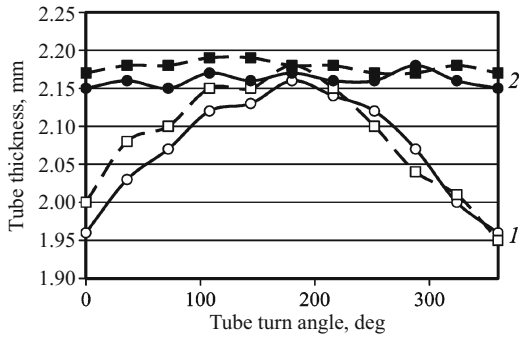

Fig. 4. Circumferential thickness variation of a type-B tube before (*1*) and after (*2*) unilateral glass deposition with burner scan speed 0.28 mm/sec and oxygen flow rate through the evaporator with $SiCl_4 \approx 2$ liters/min.

crease the circumferential thickness variation of the tube. For example, $1/v \approx 3.6$ sec/mm for a type-B tube with thickness variation 0.2 mm.

The results on eliminating the circumferential thickness variation of this tube in one pass of the burner with scan speed 0.28 mm/sec are presented in Fig. 4.

In contrast to deposition on a rotating tube the temperature distribution along the circumference with uniform deposition was nonuniform. The shrinkage density was nonuniform. The shrinkage density and the adhesion to the surface were different. The porous layer was compact on the lower part of the tube and loose on along the sides. For this reason, a thin porous layer was added on a rotating tube with burner scan speed 0.5 mm/sec in order to normalize the state of the deposition. This addition operation resulted in a uniform glassy layer.

CONCLUSIONS

The results presented above are important for applications, making it possible to increase substantially the quality of domestically produced quartz tubes with respect to the geometric parameters and the purity of the glass in the surface layer.

This work was performed at National Research University ITMO with the financial support of the Ministry of Education and Science of the Russian Federation (project No. 02.G25.31.0044).

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